

PAT 34 ANDT
AS ENCLOSED TO IPER

We claim:

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1. A process for preparing methyl formate by reacting excess methanol with carbon monoxide under superatmospheric pressure and at elevated temperature in the presence of alkali metal methoxide or alkaline earth metal methoxide as catalyst in a pressure-rated reactor, separating the methyl formate formed from the reaction product and recirculating the liquid phase which is essentially free of methyl formate to the reactor, with part of the liquid phase to be recirculated being discharged and fresh catalyst solution being fed in, wherein

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the reaction is carried out in a cascade comprising at least two reactor elements

at from 80 to 120°C,

under a carbon monoxide pressure of from 90 to 180 bar,

in the presence of from 0.05 to 0.5% by weight, based on the weight of the liquid feed, of an alkali metal alkoxide or alkaline earth metal alkoxide,

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the ratio of the amounts of starting materials fed in per unit time, the reaction temperature, the pressure and the residence time of the reactants in the reactor elements are set so that at least that amount of methanol required to keep both the catalyst used and its degradation products virtually completely dissolved under the reaction conditions in the reactor and in the fresh reaction product remains unreacted,

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the total output from the reactor is passed to a stripping apparatus in which essentially the methyl formate is stripped from the reaction mixture,

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a part TR of the remaining liquid phase is recirculated to the reactor and a part TA is discharged, with the TR:TA split controlled as a function of the alkali metal formate or alkaline earth metal formate content of the degassed reaction product so that solid precipitates of alkali metal salts or alkaline earth metal salts occur at no point in the process,

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and residual catalyst and catalyst degradation products are removed solids-free from the discharged part in a desalting apparatus and the remaining methanol is returned directly or indirectly to the reactor.

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2. A process as claimed in claim 1 carried out using from 2 to 5 reactor elements.

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3. A process as claimed in claims 1 and 2, wherein steam and/or hot water and, if desired, additional heat are fed to the discharged part TA of the liquid phase remaining after separating off the methyl formate, consisting essentially of methanol containing catalyst and catalyst degradation products, in the desalting apparatus in such amounts that the methanol is essentially completely vaporized and an aqueous solution of the catalyst degradation products is obtained.

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4. A process as claimed in claims 1 to 3, wherein the desalting apparatus is operated as an integrated heat system with the distillation apparatus and the methanol vapor leaving the top of the desalting apparatus is fed to the distillation apparatus.

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5. A plant for producing methyl formate by the process of claim 1, comprising

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A) a synthesis group consisting essentially of

5 A1) a reactor having at least two separately heatable and coolable reactor elements with feed lines for fresh methanol, recirculated catalyst-containing methanol for fresh methanolic catalyst solution and for a gas mixture comprising carbon monoxide, at least one outlet line each for the reaction product and residual gas, devices for generating and maintaining a fine dispersion of the gas stream in the liquid stream, and instrumentation for monitoring temperature and pressure,

10 A2) a depressurization apparatus provided with cooling elements for depressurizing the reaction product to the work-up pressure and provided with a feed line for the reaction product and outlet lines for residual gas and liquid phase,

15 B) a work-up group consisting essentially of

20 B1) a distillation apparatus for separating essentially methyl formate from the liquid phase of the reaction product provided with a feed line for the liquid phase and outlet lines for essentially methyl formate and for remaining catalyst-containing methanol,

25 B2) an adjustable stream divider for dividing the methanol stream containing residual catalyst and catalyst degradation products leaving the distillation apparatus into the substreams TR and TA,

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B3) a desalting apparatus which may be provided with heating and cooling elements and operates in a solids-free manner, provided with inlets for methanol containing residual catalyst and catalyst degradation products and for hot water or steam and outlets for methanol vapor and for aqueous salt solution,

C) connection lines and, if required, pumps for appropriate transport of reaction participants and products between the elements of the plant sections A and B and feed lines for starting materials and outlet lines for methyl formate and waste gas.

6. A plant as claimed in claim 5, wherein the distillation apparatus B1 used is a column whose separation efficiency is sufficient for methyl formate of the required purity to be taken off from the degassed reaction product via the top.

7. A plant as claimed in claims 5, 6 and 7, wherein the desalting apparatus B3 consists essentially of a rectification column which can be heated or cooled in zones if necessary, may be provided with separation plates and is equipped in suitable positions with feed lines for methanol containing residual catalyst and catalyst degradation products and for hot water and/or steam, outlet lines for taking off distillate at the top and water containing catalyst degradation products from the bottom, and whose separation efficiency is sufficient to take off methanol containing less than 100 ppm, preferably less than 30 ppm, in particular from about 5 to 15 ppm, of water at the top.

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8. A desalting apparatus consisting essentially of 1) a rectification column which can be heated or cooled in zones if necessary, maybe provided with separation plates whose separation efficiency is sufficient to take off methanol containing less than 100 ppm of water at the top, 2) which is
5 equipped in suitable positions with a feed line for methanol containing residual catalyst and catalyst degradation products, 3) which is equipped with a feed line for hot water and/or steam, and 4) outlet lines for taking off distillate at the top and water containing catalyst degradation products from the bottom.
- 10 9. A combination of a distillation apparatus B1 and the desalting apparatus of claim 8 in which the methanol vapor leaving the top of the desalting apparatus is conveyed via a connecting line or an equivalent integrated construction to the distillation apparatus as heat exchange medium.